

6. SPECIAL FACILITIES

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6. SPECIAL FACILITIES

6.1 OVERVIEW

Two facilities that are used in research programs at EML are described in this section: Radon Gas and Radon/Thoron Progeny Facilities for Testing and Research, and the Scanning Electron Microscope Facility. These facilities are stationary and are located in EML's quarters in New York City.

6.2 RADON GAS AND RADON/THORON PROGENY FACILITIES FOR TESTING AND RESEARCH

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6.2.1 SCOPE

Two major on-site EML facilities that are used for research on Rn gas measurements, instruments, and methods are described here (the pulse ionization chamber facility and the Rn, Th, and progeny exposure facilities).

6.2.2 THE EML PULSE IONIZATION CHAMBER FACILITY FOR RADON-222 MEASUREMENTS

6.2.2.1 INTRODUCTION

The pulse ionization chamber facility is used for the direct measurement of environmental levels of ^{222}Rn in air and breath samples. The chambers are calibrated periodically by de-emanating Rn gas from Ra solutions certified by the National Institute of Standards and Technology (NIST). Thus, the facility also serves as the EML reference for Rn gas measurements, and values so obtained are considered to be EML's best estimate of the Rn activity in a sample.

The following discussion will be confined to the direct measurement of ^{222}Rn in air samples as part of an integrated calibration facility. A fuller description of the EML pulse ionization chamber facility can be found in Fisenne and Keller (1985).

6.2.2.2 PRINCIPLE OF OPERATION

The EML pulse ionization chamber is operated on the principle of fast pulse counting (Curtiss and Davis, 1943). Fast pulse counting is based on direct detection of the " ionization produced in a single ionization chamber. To accomplish fast pulse counting, it is mandatory that O_2 and water vapor be removed before a sample is introduced into the

chamber.

6.2.2.3 DESIGN

The EML pulse ionization chambers, which are shown in Figure 6.1, are constructed of stainless steel. The interior surface is electropolished to remove surface contamination and to lower the background count rate, which is the limiting factor for measurement sensitivity. Clean chambers have a background of about 10 counts h^{-1} .

The EML chambers are constructed with a plug in the baseplate to accommodate an electrodeposited standard source for the determination of the " counting plateau. The " detection efficiency of the chambers with such a source is 52%.

The transfer systems for the removal of O_2 and water vapor are designed to meet three criteria: simplicity; a small ratio of dead space to chamber volume; and dichotomy, that is, access to one or two ionization chambers.

6.2.2.4 PROCEDURE

For direct transfer and measurement to a single chamber, the air or breath sample volume is restricted to 1 L or less. Sample transfer is accomplished by evacuating the entire system, including the ionization chambers. Pure H_2 gas is added to the sample container (35 kPa of H_2). The sample is transferred through the system with forming gas (85% N_2 , 15% H_2). The gas flow rate through the system is regulated by a capillary orifice and small particles are removed by a filter paper. The added excess H_2 and the O_2 in the sample react in the platinum black catalyst cartridges to produce H_2O and excess heat. The H_2O formed in the catalytic reaction is removed in a Drierite column. The gas is collected in a single ionization chamber which is pressurized to 35 kPa with forming gas. A block diagram of the ionization chamber and external transfer apparatus is shown in Section 4, Procedure Rn-01-RC in which the details of the procedure for the measurement of air samples are described.

6.2.2.5 CALIBRATION

The EML pulse ionization chambers are calibrated with NIST standard reference material (SRM) ^{226}Ra solutions using the Rn emanation technique (see Section 4, Procedure Ra-07-RC). In April 1984, NIST issued a new series of SRM ^{226}Ra solutions for Rn emanation measurements. As part of EML's internal quality control program, a major effort was undertaken to determine the calibration factor for the nine ionization chambers currently in use. The overall mean and standard deviation of 81 emanations

into the nine chambers was 6245 ± 135 counts h^{-1} per Bq of ^{222}Rn in equilibrium with its short-lived progeny. This value was in excellent agreement with previous calibrations.

6.2.2.6

ROUTINE CHAMBER CHECKS AND MAINTENANCE

The backgrounds of the nine pulse ionization chambers are measured with forming gas each weekend and occasionally during the work week to ensure against temporal biases. The backgrounds for the nine chambers in service range from 8-16 counts h^{-1} . A control chart of the weekend background count rates is maintained for each chamber. At the beginning of the calendar year, the yearly average and running average background rates from previous years are calculated and control limits are established for the year.

Over a period of years, the background of any chamber increases due to the buildup of long-lived Rn progeny on the interior surfaces. The background increase is a function of exposure in terms of ^{222}Rn Bq h^{-1} in the chamber, that is, 0.037 Bq of ^{222}Rn in a chamber for 17 h will produce 2.2×10^{-6} Bq of ^{210}Pb . After 1 y, the ^{210}Po will have reached 82% of the ^{210}Pb activity and will contribute an additional 3×10^{-3} counts h^{-1} to the chamber background. Fractions or multiples of the .037 Bq of ^{222}Rn example are additive in the total temporal increase of the chamber background count rate. The background count rate is reduced by dismantling and electropolishing the chamber.

The platinum black catalyst and Drierite are kept free of water vapor by maintaining these cartridges under vacuum, except during sample introduction into a chamber.

The chamber systems are checked occasionally for electrically generated noise by filling the chambers with room air. The O_2 in the air effectively reduces the pulse sizes below the 0.75 V tripping level of the electronic system and only electrically generated pulses are registered during the overnight measurement period. The electrical noise in the chamber system is < 0.25 counts h^{-1} .

Throughout the year the calibration factor of each chamber is checked by emanating Rn from a standard Ra emanation flask.

6.2.3 RADON, THORON, AND PROGENY EXPOSURE FACILITY

6.2.3.1 INTRODUCTION

The EML radon, thoron, and progeny exposure facility consists of a walk-in chamber that is used for research, testing, calibration, and for the evaluation of measuring instruments.

The 30 m³ chamber, installed in 1993, provides for a well-controlled, clean, airtight, and uniform test environment. Research with inert aerosols and other pollutants can also be carried out. Figure 6.2 shows the major features of the exposure facility.

6.2.3.2 DESCRIPTION OF THE EXPOSURE FACILITY

A. Radon and thoron gas sources.

Radon is generated from a Pylon (Model RN-1025, Ottawa, Ontario, Canada) ²²⁶Ra source with 3810 kBq of radium. The source is sealed hermetically inside a container located on top of the chamber. The injection of radon into the mixing chamber is computer controlled to obtain the desired concentrations of radon inside the main chamber.

Thoron is generated from a Pylon (Model TH-1025) ²²⁸Th source. The strength of the source was 1960 kBq at the time of acquisition. The source has to be recharged every 5 yr because of its short half-life. The thoron generator can be installed in different locations inside the main chamber in order to achieve the desired airborne thoron concentrations.

B. Design characteristics of the chamber.

A schematic diagram of the chamber is shown in Figure 6.2. The interior dimensions of the main exposure room are 3.3 m x 3.83 m x 2.59 m with a total volume of 30.75 m³. The wall panels are made of enameled aluminum on the exterior, and 22 gauge stainless steel on the inside. The floor is constructed of 16 gauge stainless steel. The floors, walls, and ceiling are insulated to ensure minimum heating and cooling requirements.

The mixing room has a volume of 3.5 m³. The anteroom, which has a volume of 5.8 m³, serves as a means to transfer instruments in and out of the main chamber and as a

buffer between the main chamber and the adjacent laboratory space. Two viewing windows, with triple panes, are located on the west wall. On the same wall there are 10 sampling ports that are 10 cm in diameter at a height of 1 m off the floor. A two-way audio system is provided for communication purposes.

C. Environmental control characteristics.

The environmental conditioning system (Fidelity Engineering, Hunt Valley, MD) consists of the following systems: refrigeration, heating, humidification, dehumidification, air delivery, control, and pressurization air.

The refrigeration system consists of a single air-cooled condensing unit, two refrigeration evaporator coils, and a refrigerant piping network. The cooling coils are made of copper with aluminum fins; the refrigerant is HCFC-22. All of the elements of the air conditioning system are constructed to minimize contamination. The compressor, condenser coils receiver, and filter dryer are located outside the chamber, adjacent to the mixing room (see Figure 6.2). The evaporator coils are inside the mixing room. The temperature in the main test chamber can be controlled from 5°C - 40°C. The difference in temperature anywhere within the chamber is no more than 1°C.

For humidification of the environment, water vapor is generated by an evaporative type system which is installed inside the mixing room. The system runs hot water across a screen-like surface and allows the water to evaporate into the air. The temperature of the water is kept below boiling. Dehumidification is accomplished by a refrigeration coil that becomes active only on call. It is isolated by a damper for the defrost cycle to avoid reintroduction of humidity during defrosting. The humidity is variable between a minimum that is determined by a -10°C dew point temperature and a maximum of 95%. The humidity inside the chamber is controlled to within 2 units of the set point during steady conditions.

The environmental conditioning system also has a hydronic hot water system located outside the test chamber. It consists of a small glass electric hot water heater of about 2000 W, a water circulation pump, a diaphragm expansion tank, and a filter. The hot water system is used to heat the test chamber air as needed.

The air delivery system, located in the mixing room, uses an in-line axial fan that can deliver a maximum of 30 m³ min⁻¹. A typical operating range is 3-10 m³ min⁻¹. In addition, a filtered pressurization air system is used to create a slight positive pressure in the main chamber to prevent uncontrolled infiltration.

D. Aerosol generator systems.

A TSI Model 3470 condensation monodisperse aerosol generator (TSI, St. Paul, MI) located outside the main chamber, is used to produce particles of the desired concentration and size. Vaporized Carnauba wax condenses on NaCl nuclei to produce monodisperse aerosols with a geometric standard deviation (F_g) of around 1.1 or less. The concentration of the aerosols inside the test chamber can be controlled from 1,000 to 30,000 cm^{-3} . If polydisperse aerosols are desired, the concentration can be increased to more than 100,000 cm^{-3} . The generated aerosols are injected through a sampling port hole into the main chamber. Aerosols from a burning candle, an electric heater, a kerosene lamp, or from cigarette smoke, can be generated individually or in combination by placing the generators inside the test chamber. The particle concentration inside the main chamber without any generated aerosol is $< 200 \text{ cm}^{-3}$.

E. Aerosol and vapor monitoring systems.

The aerosol concentration inside the main chamber is measured with an Environment One Rich Model 200 condensation nucleus counter, or a TSI Model 3025 ultra-fine particle counter. The size distribution of the test aerosol is measured with a TSI scanning mobility particle sizer. A Bruel & Kjaer Multi-gas Monitor Type 1302 is used to measure key organic pollutants. The detection limit depends on the type of pollutant and ranges from 0.001 - 1.0 ppm.

F. Radon control and monitoring systems.

The concentration of radon, thoron and progeny is regulated by adjusting the output from the radon generator. Radon is measured with two continuous scintillation cell monitors (cell volume = 0.096 and 3.3 L). The monitors are interfaced to a computer located at the control panel outside the chamber. The calibration and the accuracy of the radon monitors are based on intercomparisons made with pulse ionization chambers (see Section 6.2.2). The data are logged to a computer at 15-min intervals, and may be recalled at any time. Radon and thoron concentrations in the main chamber are adjustable in the range from 100 - 5000 Bq m^{-3} , and 50 - 5000 Bq m^{-3} , respectively.

The radon and thoron progeny concentrations are regulated by the dilution of the parent gases and by the presence of aerosols. The progeny concentrations are measured inside the main chamber by grab, integrating, and continuous monitoring devices. The standard monitoring instruments for radon and thoron progeny are the AlphaSmart-760 (Alpha Nuclear, Mississauga, Ontario, Canada) and the GRI-1100 monitor. The analyses for the individual radon and thoron progeny and the potential alpha energy concentrations are performed using the methods of Thomas (1972), Nazaroff (1983), and Raabe and Wrenn (1969). All three methods are computerized and the data are available on an hourly basis. The concentrations in the main chamber range from $5.6 \times 10^{-9} \text{ J m}^{-3}$ to $1.4 \times$

10^{-5} J m^{-3} for radon progeny, and 1×10^{-5} to $7 \times 10^{-5} \text{ J m}^{-3}$ for thoron progeny.

Because of the capability to maintain the test chamber at less than $200 \text{ particle cm}^{-3}$, the equilibrium factor as low 0.01 is achievable. At very high particle concentrations, an equilibrium factor of 0.5 is obtainable.

The sensors for temperature and humidity are located in the main test chamber and in the mixing room. The static pressure in the main chamber and the speed of the conditioned air exiting from the mixing room are monitored continuously. The data are automatically logged on the control computer.

G. Radon progeny particle size distribution measurements.

The test chamber particle size conditions can be measured with the micro-orifice uniform deposit impactor (MOUDI - see Section 2.2.2.5), EML developed screen diffusion batteries (see Section 2.2.2.6), and graded screen array (see Section 2.2.2.7). When used in combination, these instruments can measure radon and thoron progeny particle sizes ranging from 0.5 to 5000 nm (Tu and Knutson, 1994).

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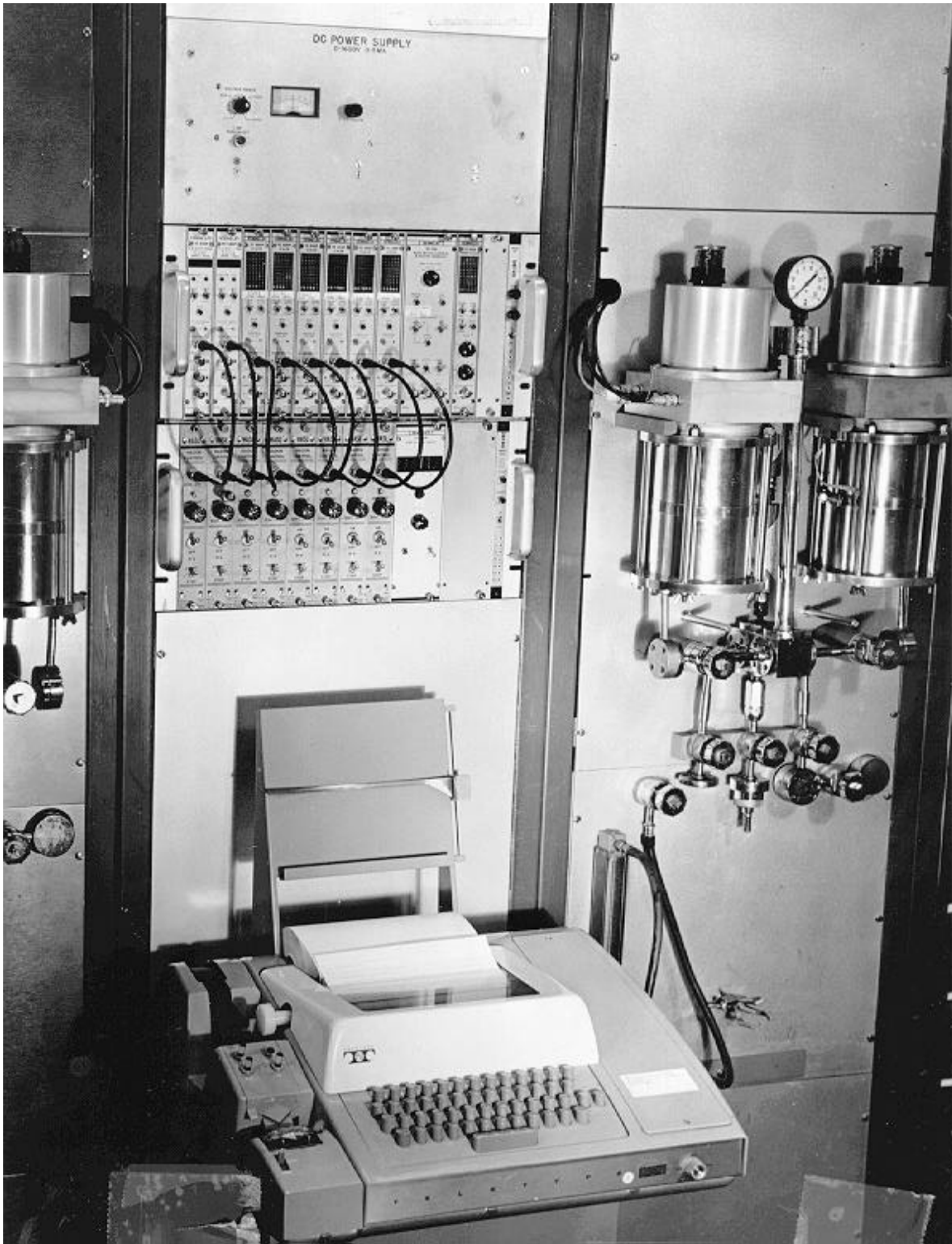


Figure 6.1 Photograph of pulse ionization chamber facility.

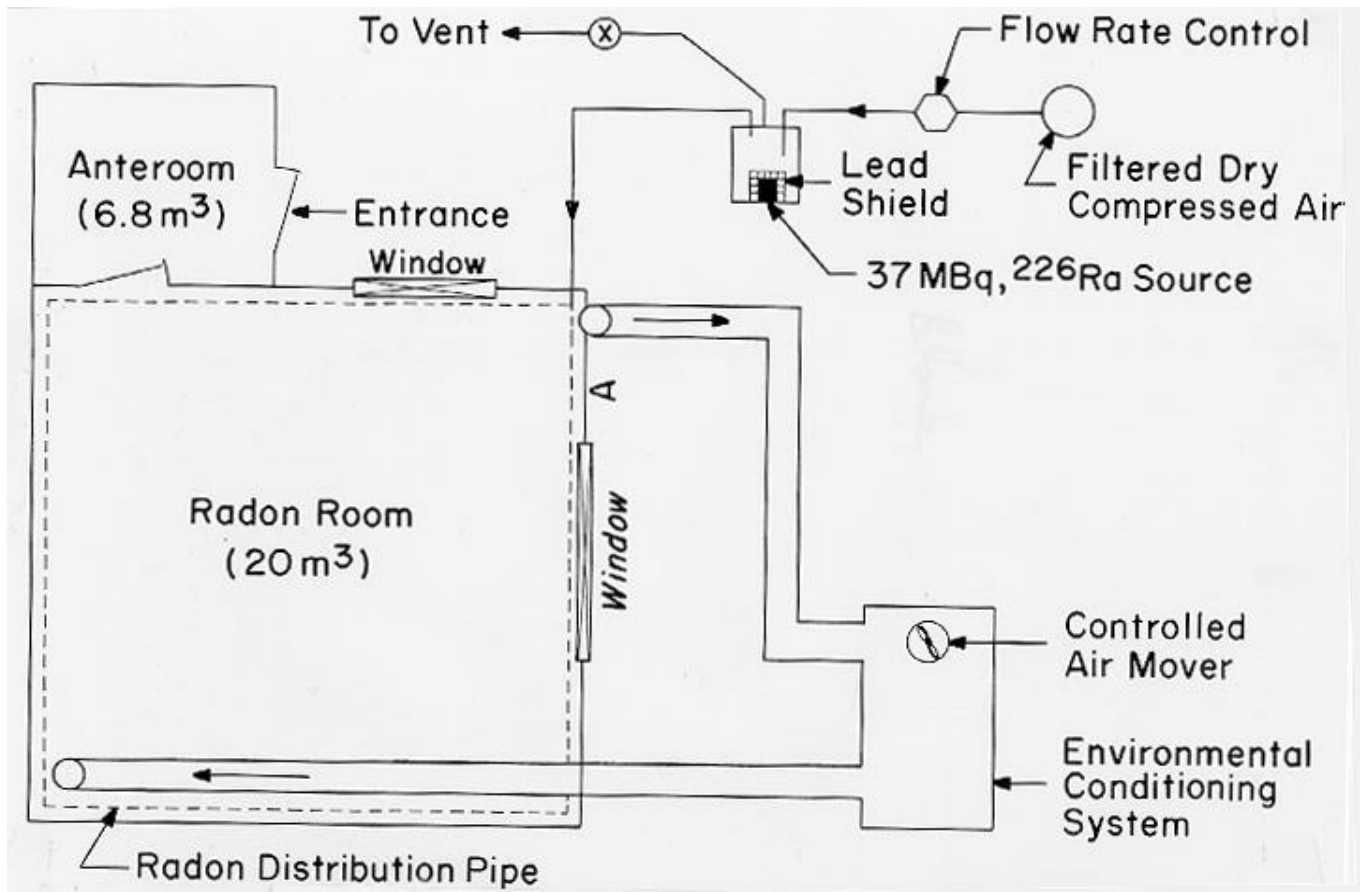


Figure 6.2 Schematic drawing of radon/thoron and progeny exposure facility.

6.3 SCANNING ELECTRON MICROSCOPE FACILITY

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6.3.1 SCOPE

This section describes EML's scanning electron microscope (SEM) facility. The physical layout of this facility is shown in Figure 6.4. The SEM is used to: 1) analyze aerosols collected from the troposphere and the stratosphere (Kromidas et al., 1996); 2) characterize laboratory-generated aerosol; and 3) analyze biological samples.

6.3.2 SAMPLE PREPARATION ROOM

The sample preparation room is maintained under clean-room conditions using high efficiency particulate air (HEPA) ceiling filters. The air is recirculated and controlled with a system that is independent of the building air delivery system. There are two Class-100 benches to provide additional cleanliness for sample preparation and for the preparation of sampling equipment. The first, an exhausting laminar bench (Advanced Purification Inc., Hauppauge, NY 11788; Model G68-PD) allows the use of various solvents and provides a Class 100 environment for equipment setup and preparation of biological samples. The second bench (Laminar Flow Inc., Ivyland, PA 18974; special order) is a recirculating vertical laminar flow system with two cartridges that contain molecular sieve 5A and activated charcoal. These cartridges remove substances from the airstream that may interfere with aerosols. Such interfering substances include ammonia, water vapor, and organics.

6.3.3 MICROSCOPE ROOM

The microscope room is also maintained under clean-room conditions using HEPA ceiling filters.

6.3.3.1

SCANNING ELECTRON MICROSCOPE

The SEM is an AMRAY-1820 (AMRAY, Bedford, MA 01730) and has a 150-mm specimen chamber and a resolution of 5 nm. The SEM may be operated at low voltages (~1 kV), thus allowing imaging of samples without prior preparation. Microprocessor

controlled operations permit automatic image focusing and stigmatism and provides push button digital magnification from 25X- 400,000X at a 12 mm working distance. However, just how much of this magnification is usable depends heavily on the contrast characteristics of the sample. The SEM is supplied with a solid-state backscatter detector. This detector produces images which emphasize maximum atomic number differences. The resulting image shows contrast differences that can be related to differences in trace element composition. The SEM is also supplied with an X-ray/imaging system (described below). This system controls the sample stage automatically and thus allows for unattended X-ray analyses, imaging, and sample characterization of more than 100 different fields.

The whole microscope assembly is isolated on a Micro-g vibration isolation table (TMC, Peabody, MA 01960).

6.3.3.2 MICROANALYSIS SYSTEM

The microanalysis system used in conjunction with the AMRAY 1820 SEM is the PGT IMIX system with a two position light element Si(Li) energy dispersive spectrometer (Model 4200 EDS, Princeton Gamma-Tech, Princeton, NJ 08540). The PGT IMIX system is equipped with:

1. An ultrahigh resolution 19-in color display.
2. Dec 11/73 high performance processor with floating point.
3. SPARC station 1+ (SUN Microsystems) with a built-in, 3 1/2" floppy disk drive, external CD ROM drive, and a 1.27 gigabyte Fujitsu drive.
4. Two thousand channel data collection system.
5. Pulse processing electronics.
6. EDS X-ray analysis software.
7. Qualitative and quantitative analysis software.
8. Chemical classification software.
9. PGT beam control package.
10. Advanced feature analysis package.

11. Mega plane option (800 x 1040 pixel image).

The microanalysis system allows detailed chemical and size classifications of samples. In the windowless mode, the model 4200 EDS allows the analysis of X-rays with energies down to 700 eV. In this position, detection of B, C, N, and O is possible, as well as enhanced detection of F, Na, Mg, and Al. The PGT beam control package allows automated analysis of specific points on a collection surface by controlling the scan coils of the microscope column. Up to 300 points can be automatically stored for later use.

The PGT imaging system is connected to the Tissue Culture Facility via a video cable that is attached to a CCD video camera and can capture images from the optical microscopes.

6.3.3.3

SAMPLE CRITICAL POINT DRYING AND COATING EQUIPMENT

The sample coating equipment consists of a Denton high vacuum evaporator (Denton Vacuum, Inc., Cherry Hill, NJ 08003), an EFFA Carbon Coater (Ernest E. Fullam Co., Latham, NY 12110; Model No. 12560), and a sputter coater (Polaron, Warrington, PA; Model E5000). The sputter coater is supplied with platinum, gold, and silver targets. The carbon coater and sputter coater, plus an optical microscope are housed in a vertical flow class 100 laminar clean bench (Laminaire Corp., Rahway, NJ 07065; Model DWS-630).

Critical point drying of biological samples is accomplished via the Balzers CPD 030 using acetone as the transfer medium and liquid CO₂ as the transition liquid. The CPD 030 is located in the Sample Preparation Room with access to the exhaust laminar flow bench.

6.3.4 QUALITY CONTROL

When using an SEM, several things should be kept in mind. The sampling process must be conducted very carefully to provide the microscopist with a representative sample of the material of interest. Many methods of sampling introduce bias in that their collection efficiency varies with particle size. The internal environment of the microscope should also be taken into account. In order to function, the column is kept at a high vacuum (10^{-5} Torr) and there can be large amounts of heat generated in the sample by electron bombardment. These conditions can alter or destroy susceptible samples. Contamination is always a concern. Considering the size of the particles of interest, unless strict sample handling procedures are followed, contributions from contaminating

sources may outweigh the sample material.

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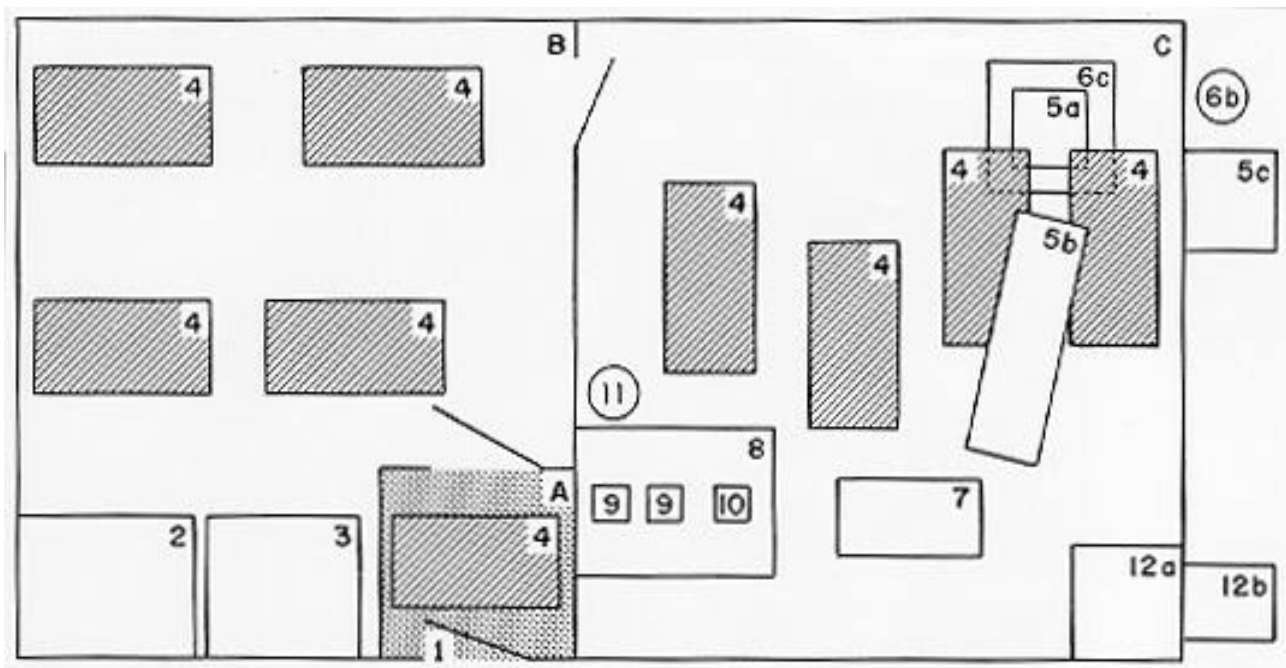


Figure 6.3 SEM facility: A) clothing change room; B) sample preparation room; C) SEM room. Areas in rooms: 1) sticky floor mat; 2) exhaust laminar flow bench; 3) recirculating laminar flow bench; 4) HEPA ceiling filters with blowers; 5a) column and stage; 5b) SEM electronic console; 5c) SEM vacuum, air and water support module (equipment placed outside microscope room); 6a) Micro-g vibration table; 6b) air supply for Micro-g table; 7) PGT microanalysis system; 8) vertical laminar flow bench; 9) particle coating devices; 10) optical microscope; 11) tank of Argon; 12a) Denton high vacuum evaporator; 12b) evaporator and coating device vacuum, air and water module.